
गुरुत्वाकर्षण तरल अवसादन विधियों द्वारा
कण आकार वितरण का निर्धारण
भाग 3 एक्स-रे गुरुत्वाकर्षण तकनीक
(पहला पुनरीक्षण)

**Determination of Particle Size
Distribution by Gravitational
Liquid Sedimentation Methods
Part 3 X-Ray Gravitational Technique
(First Revision)**

ICS 19.120

© BIS 2023



भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुर शाह ज़फर मार्ग, नई दिल्ली - 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI - 110002
www.bis.gov.in www.standardsbis.in

August 2023

Price Group 6

FOREWORD

This Indian Standard (Part 3) (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Sieves, Sieving and Other Sizing Methods Sectional committee had been approved by the Civil Engineering Division Council.

Gravitational sedimentation particle size analysis methods are based on the settling velocity, under a gravitational field of particles in a liquid. This method is widely used for determining size distribution of many particulate materials. Typically, the gravitational methods apply to samples in the 0.5 μm to 100 μm size range and where the sedimentation condition for a Reynolds number < 0.25 is satisfied.

The choice of the most suitable method for determination of size distribution depends upon factors like:

- a) the purpose for which the analysis is required;
- b) the size range and density of particles;
- c) the important properties of powder;
- d) the amount available for test; and
- e) the method by which the gross sample has been collected.

Due to the inherent characteristics of the different materials and the factor stated above, it is not possible to apply a single method of size analysis. This standard recommends procedures that may be applied in the majority of cases. The purpose of this standard is to provide uniformity in procedure for any gravitational method selected to facilitate comparisons of size analysis data.

This standard was first published in 1969 with the title 'Liquid sedimentation methods for determination of particle size of powders'. In this revision, to incorporate the latest advancement in gravitational liquid sedimentation methods for determining size distribution of particulate materials, the gravitational liquid sedimentation method has been grouped into four parts.

This standard (Part 3) describes a method for the determination of the particle size distribution of a powder dispersed in a liquid using gravity sedimentation. The measurement of the concentration of solids settling in a liquid suspension is achieved by monitoring the incremental signal absorption from a beam of X-rays. The other parts in the series are:

- Part 1 General principles and guidelines
- Part 2 Fixed pipette method
- Part 4 Balance method

In the formulation of this standard due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the field in this country. This has been met by deriving assistance from the following publications:

ISO 13317 (Part 1) Determination of particle size distribution by gravitational liquid sedimentation methods — Part 1: General principles and guidelines

ISO 13317 (Part 2) Determination of particle size distribution by gravitational liquid sedimentation methods — Part 2: Fixed pipette method

ISO 13317 (Part 3) Determination of particle size distribution by gravitational liquid sedimentation methods — Part 3: X-ray gravitational technique

ISO 13317 (Part 3) Determination of particle size distribution by gravitational liquid sedimentation methods — Part 4: Balance method

The composition of the Committee responsible for the formulation of this standard is given in Annex B.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*). The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Indian Standard***DETERMINATION OF PARTICLE SIZE DISTRIBUTION BY
GRAVITATIONAL LIQUID SEDIMENTATION METHODS****PART 3 X-RAY GRAVITATIONAL TECHNIQUE***(First Revision)***1 SCOPE**

1.1 This standard (Part 3) covers X-ray gravitational technique for the determination of the particle size distribution of a powder dispersed in a liquid using gravity sedimentation. The measurement of the concentration of solids settling in a liquid suspension is achieved by monitoring the incremental signal absorption from a beam of X-rays.

1.2 The methods of determining the particle size distribution described in this standard are applicable to powders which can be dispersed in liquids or powders which are present in slurry form. The typical particle size range for analysis is from about 0.5 µm to about 100 µm. The method is used for materials containing particles of the same chemical composition which produce adequate X-ray opacity.

2 REFERENCES

The standards given below contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication, the edition indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of the standard:

<i>IS No.</i>	<i>Title</i>
IS 4124 : 1981	Glossary of terms relating to powders (<i>first revision</i>)
IS 5282 (Part 1) : 2023	Determination of particle size distribution by gravitational liquid sedimentation methods: Part 1 General principles and guidelines (<i>first revision</i>)
IS 8883	Methods of sampling chemical and chemical product:
(Part 1) : 2005	General requirements and precautions (<i>first revision</i>)
(Part 2)	Sampling equipment,
(Sec 1) : 1978	For solids
(Sec 2) : 1978	For liquids
(Sec 3) : 1978	For gases
IS 18100 : 2022	Sample preparation — Dispersing procedures for powders in liquids

3 TERMS AND DEFINITIONS

For the purpose of this standard, the definitions given in IS 4124 and IS 5282 (Part 1), and the following shall apply.

4 SAMPLING

The sampling shall be done as the method given in IS 5282 (Part 1).

5 PRINCIPLE

The concentration of dispersed sample is measured by attenuation of an X-ray beam. A stable, narrow, collimated beam of X-rays passes through a suspension of the sample: it is detected at a known height from the top of the sample cell. The sample cell is completely filled with sample suspension for the duration of the analysis. The settling height, h , at which the particle concentration, C , is determined, may be reduced during the analysis for the purpose of obtaining a more rapid analysis than would be possible if all measurements were made at the same value of, h . The cumulative mass percent of sample present at a given sedimentation height is continuously determined. The X-ray signal attenuation at the known height is compared to the attenuation with suspending liquid and also to the attenuation with the homogeneously dispersed sample present in the liquid. The attenuation of the emergent X-ray beam is proportional to the mass of powder in the beam, and is expressed by the following formula:

$$I = I_0 \exp(-BC) \quad \dots \quad (1)$$

or

$$C = (-1/B) \ln(I/I_0) \quad \dots \quad (2)$$

The X-ray density, D , is expressed as follows:

$$D = -B C l_{ge} \quad \dots \quad (3)$$

also

$$D = \lg(I_0/I) \quad \dots \quad (4)$$

thus

$$D = -B C l_{ge} = \lg(I_0/I) \quad \dots \quad (5)$$

The Stokes diameter x_{St} , corresponding to the X-ray density at settling height h and time t , is given by

$$x_{St} = \left[\frac{18\eta h}{(\rho_s - \rho_l)gt} \right]^{1/2} \quad \dots \quad (6)$$

The X-ray density D is proportional to the concentration C , and thus mass, of sample in the beam. A plot of the X-ray density D , taken as a function of time t and h , versus

$$\sqrt{18\eta h / (\rho_s - \rho_l)gt}$$

provides the cumulative mass distribution versus equivalent spherical diameter.

where

I = Attenuation of emergent X-ray beam through suspension at settling height h , at time t ;

I_0 = Attenuation of emergent X-ray beam passing through the suspending fluid;

B = Function of atomic number of sample elements in beam;

C = Concentration of sample in beam;

D = X-ray density [$\lg_{10} (I_0/I)$];

x_{St} = Stokes equivalent spherical diameter in m or μm ;

h = Sedimentation height in m; and

t = Sedimentation time in s;

η = Suspending liquid viscosity in Pa·s or mPa·s;

ρ_s = Sample density [effective particle density] in km. m^{-3} ;

ρ_l = Liquid density in km. m^{-3} ; and

g = Acceleration due to gravity in m. s^{-2} .

6 APPARATUS

6.1 Main Apparatus

The main apparatus (*see* Fig. 1) typically consists of a temperature-controlled analysis compartment and mixing chamber; a plumbing system for circulation of suspending liquid or sample suspension; an X-ray source/detector system and a control module for apparatus control, data acquisition and reduction. The sedimentation cell within the temperature-regulated analysis compartment may be repositioned throughout the analysis relative to the signal source and detector to reduce analysis time. Alternative designs, such as one in which the X-ray source and detector move while the sedimentation cell remains stationary, may also be used.

The use of a magnetic stirrer should be avoided if magnetically susceptible particles are to be tested. The dispersion may then be maintained by means of a mechanical stirrer.

6.2 Ancillary Apparatus

Ultrasonic bath, probe or high-speed mechanical stirrer capable of 500 revolutions to 1 000 revolutions per minute.

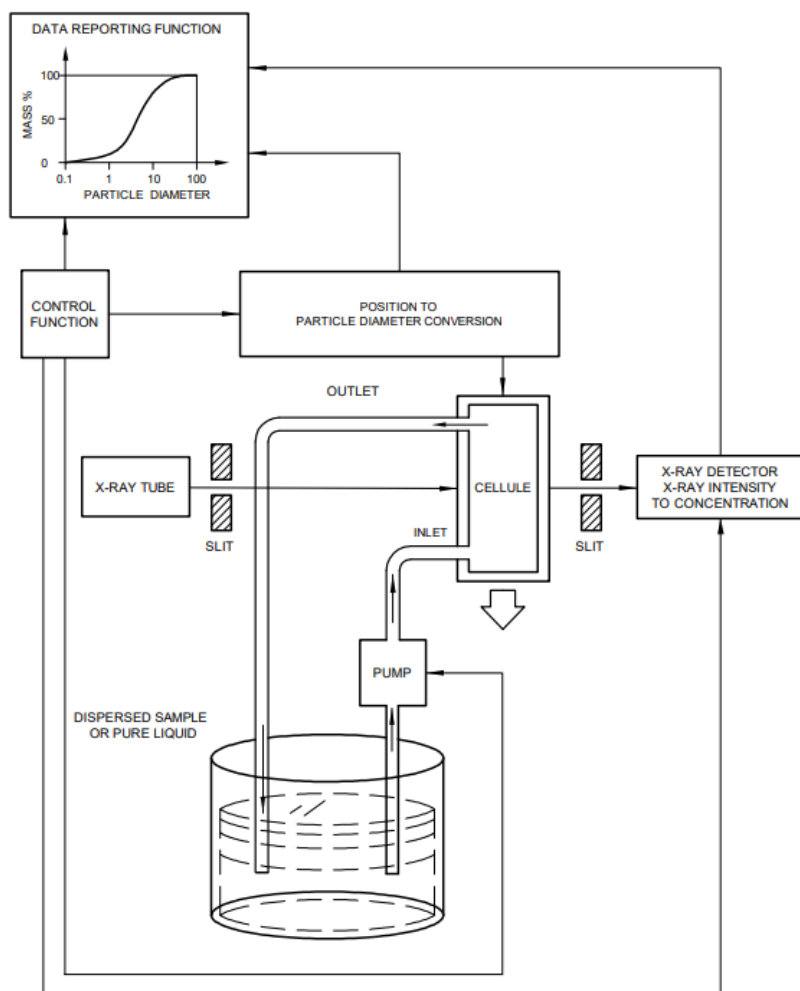


FIG. 1 SCHEMATIC OF TYPICAL APPARATUS USING THE X-RAY GRAVITATIONAL TECHNIQUE

7 SAMPLE PREPARATION

A representative sample for analysis shall be taken according to IS 8883 or any other suitable method. It shall be dispersed as per IS 18100 'Sample preparation — Dispersing procedures for powders in liquid' in a suspending medium of adequate viscosity and X-ray transparency. The use of dispersing agents and ultrasonics or mechanical stirring to aid dispersion will be recorded. Typically 50 ml of dispersed sample is required for the analysis. The sample concentration will be prepared in accordance with the instructions supplied with sample. Higher sample concentrations may be required with powders having a low X-ray absorptivity coefficient. Knowledge of the exact sample concentration is not required, but the influence of concentration should be checked. The minimum concentration of suspension compatible with the measuring method is preferred [see IS 5282 (Part 1)]. The required sample concentration will typically produce a

reduction in the beam signal of 10 percent to 30 percent relative to the signal observed with the suspending liquid. The sample may be dispersed in either an aqueous or an organic medium. Any liquid compatible with sample cell materials and having low absorptivity for X-rays may be used. Typical liquids are water, glycols, kerosene, mineral oils, alcohols and mineral spirits. It is recommended that the viscosity of the suspending medium should have a value such that the largest particle to be measured has a Reynolds number of ≤ 0.25 [see IS 5282 (Part 1)]. Any temperature change of the suspension should be minimized during a measurement (see 8.3).

The viscosity and density of the suspending liquid at the temperature of the analysis will be reported. The effective particle density of the sample is also reported. The suspending liquid, including dispersants at the analysis concentration, may be used as the displacement fluid to experimentally determine the effective particle density.

8 PROCEDURE

8.1 General

Refer IS 5282 (Part 1) for guidance on restrictions of upper size limit and lower size limit, and on test conditions. The validity of the measurement will be dependent on the Stokes equation applying to creeping flow for the suspension under study. The lower size limitation will in part be a function of the ability of the system to minimize thermal convection and mechanical effects. Additionally, for the fine size fraction, Brownian motion effects may contribute to the vertical spread of particles of the same size originating from the same initial settling height.

8.2 Base-Line Determination

Determine the attenuation of the X-ray signal with only suspending liquid in the sample cell (zero percent solids). Where possible, a base-line scan of the entire measuring portion of the cell is advised for subsequent correction of the corresponding sample suspension data. The base-line scan of the entire measuring portion of the cell is recommended as this will provide optimum correction for variability in the cell window thickness, or non-parallel alignment of the cell windows.

8.3 Temperature Equilibration

Place the dispersed sample suspension in the mixing chamber, and circulate the suspension through the sample cell for typically 60 s to 90 s. Confirmation is obtained that the analysis compartment is operating within the specified temperature band, preferably within ± 1 K of the temperature set point.

8.4 Bubble Elimination

Check for the presence of air bubbles in the sample cell either manually or automatically. If bubbles are detected, remove them by manual or automatic operation. If air bubbles are detected, make a further check prior to commencing the analysis. Repeat the bubble detection and elimination steps until confirmation is obtained that no bubbles are detected.

8.5 Concentration Setting

Whilst the suspension continues to circulate through the sample cell, determine the concentration setting, according to the manufacturer's instructions. Depending on the apparatus, this will typically require either a manual setting of the 100 percent point on the sample-recording graph paper or the automatic

detection and storage of the signal for subsequent computation. A scan of the entire measuring portion of the cell for the 100 percent concentration setting may be performed; this will provide optimum correction for variability in cell window thickness, or non-parallel alignment of the windows, when applied in combination with a similar base-line scan.

8.6 Measurement

Initiate the analysis by cessation of pumping of the dispersed sample through the sample cell. Elapsed sedimentation times and the corresponding sample concentrations, at known sedimentation heights, are automatically recorded. The cumulative signal obtained as the analysis progresses permits termination of the analysis prior to achieving a base-line corresponding to a zero concentration of detected sample. Registration of the complete cumulative size distribution is preferred for the better detection of anomalies. However, for routine procedures, termination of the measurement prior to reaching the base-line is permitted. The requirements for an abbreviated analysis will depend on the sample diameters of interest to the operator. When the measurement is completed, a repeat analysis may be performed, following adequate redispersion of the sample sediment in the sample cell.

8.7 Cleaning of Instruments

Thoroughly rinse the sample vessel, measurement cell and associated tubing with the suspending liquid following the final repeat analysis. Run a new base-line scan to determine if any sample remains from the previous analysis.

9 TESTS IN DUPLICATE, VALIDATION AND VERIFICATION

9.1 General

The X-ray gravitational technique is based on first principles and does not require calibration by the operator. Correct instrument operation may however be confirmed by a validation procedure.

9.2 Tests in Duplicate

Carry out tests in duplicate on representative analysis samples taken from the same laboratory sample. The results for duplicate analyses should normally differ by less than 2 percent for the proportions of mass at the same Stokes diameter. This figure would be expected to hold for the certified reference materials referred to in 9.3. Note that a wider tolerance may be required for other sample types with more narrow distributions.

9.3 Validation

The total measurement procedure should be considered, including sampling, sample dispersion, sample transport through the measuring zone, and the measurement itself. It is essential that the total operational procedure be adequately described in detail. Primary validation can be made with acceptable reference materials certified by a standard gravity sedimentation technique. It is recommended to use certified reference materials for validation. A record of all validation activities shall be maintained.

The response of a sedimentation instrument is considered to meet this standard if the mean values of x_{10} , x_{50} and x_{90} , coming from at least three independent measurements, are within the range of the certified values that is. the mean values together with their standard deviation. Mixtures by mass of two or more certified reference materials, having the same properties, can be applied to test the accuracy of the reported fractional quantities, the size resolution and the sensitivity for fines or coarse material. Representative sampling is of particular concern in the case of a test of mixed reference materials where fractional quantities can be very small.

The precise position of the sample cell relative to the X-ray beam is critical for an accurate measurement. Where possible, the correct positioning at the start of the analysis is confirmed by monitoring the height from the initial analysis position to that point at which intersection of the finely collimated X-ray beam occurs with the top inner edge of the metal cell (hence referred to as an 'X-ray beam-split test'). Where applicable, an X-ray beam-split test may be performed as part of each validation test measurement, to corroborate precise positioning of the movable parts of the measurement system. This test verifies or establishes the travel of the moving parts from their initial position, for a given number of repositioning steps.

9.4 Verification

The checking at regular intervals of both operator procedure and instrument performance is essential to verify the test results.

Whilst the X-ray gravitational technique does not require calibration, it is recommended that an X-ray beam-split test, when applicable, be performed regularly to corroborate precise positioning of the movable parts of the measurement system. Where practical, it is recommended that the beam-split test be incorporated as part of each test measurement. This test verifies or establishes the travel of

the moving parts from their initial position, for a given number of repositioning steps.

It is recommended that both the operator procedure and instrument performance be verified by the regular use of certified reference materials. Product samples of typical composition and particle size distribution for a specified class of products may be used, provided that their particle size distribution has been proven to be stable over time. The results should comply with previously determined data with the same precision and bias as those for the certified reference materials; otherwise a re-run of certified reference materials is indicated to verify instrument performance.

10 CALCULATION OF RESULTS

10.1 Calculation of Particle Size

Use equation (6) to calculate the equivalent Stokes diameter for the height and time at which each measurement of X-ray density is recorded. Present the result as percentage oversize or undersize.

10.2 Calculation of Cumulative Distribution by Mass

Use equation (4) to compute X-ray density, which represents the cumulative distribution by mass below the equivalent Stokes diameter corresponding to the time and height of the measurement.

11 REPORTING OF RESULTS

Results are typically tabulated (*see* Table 1) as the cumulative percentage by mass (reported to the nearest 0.1 percent) at a near logarithmic series of diameters or plotted (Fig. 1) as cumulative mass percent versus diameter (on an exponential logarithmic scale). In the case of a plot, the diameters will be placed on the abscissa and the cumulative mass on the ordinate. A work out example is given in Annex A.

The report shall state the following:

- a) Instrument type used;
- b) Sample density;
- c) Suspending liquid;
- d) Density of suspending liquid at the analysis temperature;
- e) Viscosity of suspending liquid at the analysis temperature;
- f) Temperature of the suspension during analysis;
- g) Dispersing agent and concentration of agent used;
- h) Method of dispersion, including dispersing time; and
- j) Any other information which could have an influence on the results.

ANNEX A
(Clause 11)

EXAMPLE

Sl No.	Sample	Quartz
(1)	(2)	(3)
i)	Sample density	2 642 kg.sm ⁻³
ii)	Suspending liquid	De-ionized water.
iii)	Liquid density	994 kg.m ⁻³
iv)	Liquid viscosity	0.727 mPa.s
v)	Analysis temperature	307.85 K
vi)	Dispersing agent	0.1 percent weight per volume, tetrasodium pyrophosphate decahydrate
vii)	Dispersion method	Ultrasonic probe, 1 min

Table 1 Particle Size Distribution of Quartz
(Clause 11)

Sl No.	Diameter	Cumulative mass Distribution %
	µm	
(1)	(2)	(3)
i)	50.0	99.6
ii)	40.0	99.1
iii)	30.0	99.2
iv)	25.0	99.2
v)	20.0	99.0
vi)	15.0	98.4
vii)	10.0	95.5
viii)	8.0	92.7
ix)	6.0	86.5
x)	5.0	80.4
xi)	4.0	70.3
xii)	3.0	53.8
xiii)	2.0	29.0
xiv)	1.5	14.9
xv)	1.0	3.9
xvi)	0.8	1.2
Mass median diameter: 2.8 µm		
Mass modal diameter: 2.7 µm		

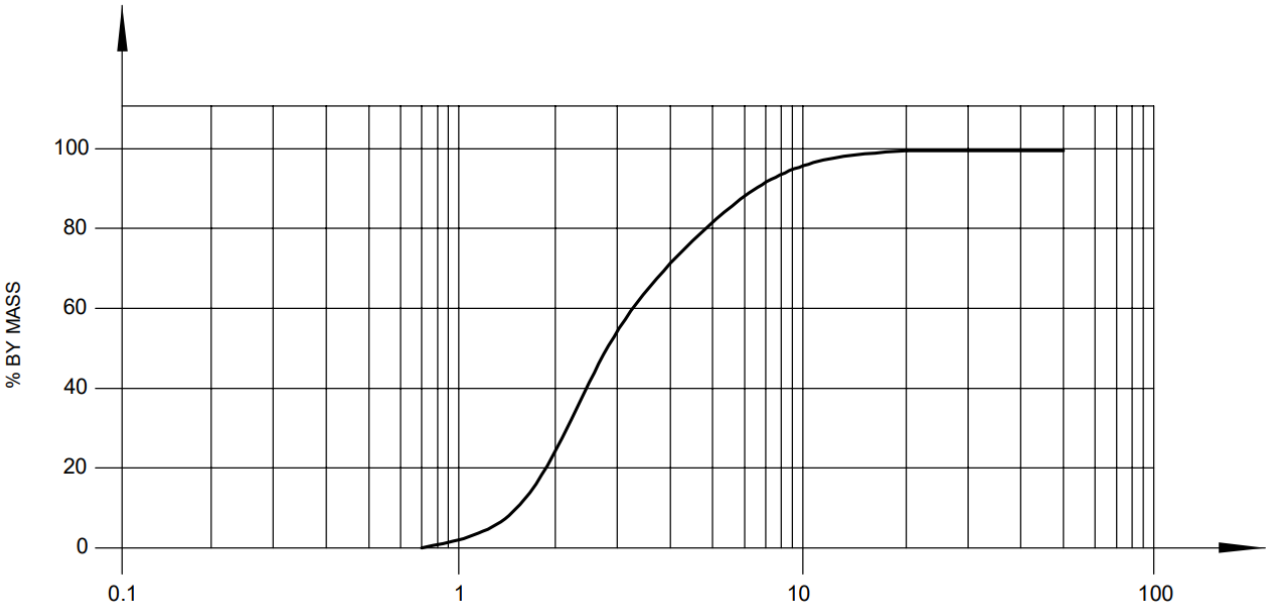


FIG. 2 PARTICLE SIZE DISTRIBUTION OF QUARTZ

ANNEX B
(Foreword)

COMMITTEE COMPOSITION

Sieves, Sieving and Other Sizing Methods Sectional Committee, CED 55

<i>Organization</i>	<i>Representative(s)</i>
In Personal Capacity (90, Savita Vihar, Vikas Marg, Delhi - 110092)	DR R. P. SINGHAL (Chairperson)
AIMIL Limited, New Delhi	SHRI ROHITASH BARUA SHRI MADAN KUMAR SHARMA (<i>Alternate</i>)
Associated Soapstone Distributing Co Pvt Ltd, Jaipur	SHRI VIKRAM GOLCHA SHRI DILIP JHA (<i>Alternate</i>)
Cement Corporation of India Limited, New Delhi	SHRIMATI SARASWATHI DEVI SHRI ANURAG KUMAR SAINI (<i>Alternate</i>)
Central Public Works Department, New Delhi	SHRI VINAYAK RAI SHRI DIVAKAR AGRAWAL (<i>Alternate</i>)
Central Soil and Materials Research Station, New Delhi	SHRI AMARDEEP SINGH SHRI B. K. MUNZNI (<i>Alternate</i>)
CSIR - Central Building Research Institute, Roorkee	SHRI A. K. SHARMA DR PRADEEP KUMAR (<i>Alternate</i>)
CSIR - National Physical Laboratory, New Delhi	DR SANJAY YADAV DR NITA DILAWAR (<i>Alternate</i>)
Haver Standard India Pvt Limited, Mumbai	SHRI DEVEN H. SHAH SHRI VIKRAM D. SHAH (<i>Alternate</i>)
Indian Bureau of Mines, Nagpur	SHRI M. G. RAUT SHRI V. A. SONTAKKEY (<i>Alternate</i>)
Indian Institute of Technology, Delhi, New Delhi	REPRESENTATIVE
Industrial Wire Netting Company, New Delhi	SHRI KESHAV GUPTA SHRI SANJAY GUPTA (<i>Alternate</i>)
Military Engineer Services, Engineer-in-Chief's Branch, Integrated HQ of Ministry of Defence, New Delhi	SHRI P. K. JAIN SHRI SOMESH KUMAR (<i>Alternate</i>)
National Council for Cement and Building Materials, Faridabad	SHRI SURESH KUMAR SHRI G. J. NAIDU (<i>Alternate</i>)
National Mineral Development Corporation Limited, Hyderabad	SHRI S. K. SHARMA SHRI G. V. RAO (<i>Alternate</i>)
National Test House, Kolkata	SHRI VIVEK SHARMA SHRIMATI MAYMOLE BOBEN (<i>Alternate</i>)
Steel Authority of India Ltd, Kolkata	SHRI BIPIN KUMAR GIRI SHRI SIBABRATA BASAK (<i>Alternate</i>)
Ultratech Cement Ltd, Mumbai	SHRI DINESH CHANDRAN K. P. SHRI DEVENDRA PANDEY (<i>Alternate</i>)

<i>Organization</i>	<i>Representative(s)</i>
In Personal Capacity (<i>B-129, F-2, Ramprastha Colony, Ghaziabad - 201011</i>)	SHRI VIRENDRA BABU
In Personal Capacity (<i>Indian Institute of Technology, Hauz Khas, New Delhi - 110016</i>)	PROF B. PITCHUMANI
BIS Directorate General	SHRI ARUNKUMAR S., SCIENTIST 'E'/DIRECTOR AND HEAD (CIVIL ENGINEERING) [REPRESENTING DIRECTOR GENERAL (<i>Ex-officio</i>)]

Member Secretary
DR MANOJ KUMAR RAJAK
SCIENTIST 'D'/JOINT DIRECTOR
(CIVIL ENGINEERING), BIS

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act, 2016* to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Head (Publication & Sales), BIS.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website- www.bis.gov.in or www.standardsbis.in.

This Indian Standard has been developed from Doc No.: CED 55 (18398).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002

Telephones: 2323 0131, 2323 3375, 2323 9402

Website: www.bis.gov.in

Regional Offices:

	Telephones
Central : 601/A, Konnectus Tower -1, 6 th Floor, DMRC Building, Bhavbhuti Marg, New Delhi 110002	{ 2323 7617
Eastern : 8 th Floor, Plot No 7/7 & 7/8, CP Block, Sector V, Salt Lake, Kolkata, West Bengal 700091	{ 2367 0012 2320 9474
Northern : Plot No. 4-A, Sector 27-B, Madhya Marg, Chandigarh 160019	{ 265 9930
Southern : C.I.T. Campus, IV Cross Road, Taramani, Chennai 600113	{ 2254 1442 2254 1216
Western : Plot No. E-9, Road No.-8, MIDC, Andheri (East), Mumbai 400093	{ 2821 8093

Branches : AHMEDABAD. BENGALURU. BHOPAL. BHUBANESHWAR. CHANDIGARH. CHENNAI. COIMBATORE. DEHRADUN. DELHI. FARIDABAD. GHAZIABAD. GUWAHATI. HIMACHAL PRADESH. HUBLI. HYDERABAD. JAIPUR. JAMMU & KASHMIR. JAMSHEDPUR. KOCHI. KOLKATA. LUCKNOW. MADURAI. MUMBAI. NAGPUR. NOIDA. PANIPAT. PATNA. PUNE. RAIPUR. RAJKOT. SURAT. VISAKHAPATNAM.